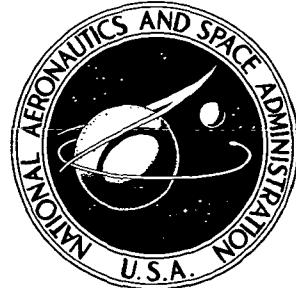


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MEMORANDUM

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HIGH-TEMPERATURE MECHANICAL
PROPERTIES OF A ZIRCONIUM-MODIFIED,
PRECIPITATION-STRENGTHENED
NICKEL - 30 PERCENT COPPER ALLOY

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SUMMARY

A precipitation-strengthened Monel-type alloy has been developed through minor alloying additions of zirconium to a base Ni-30Cu alloy. The results of this exploratory study indicate that thermomechanical processing of a solution-treated Ni-30Cu-0.2Zr alloy produced a dispersion of precipitates. The precipitates have been tentatively identified as a Ni_5Zr compound. A comparison of the mechanical properties, as determined by testing in air, of the zirconium-modified alloy to those of a Ni-30Cu alloy reveals that the precipitation-strengthened alloy has improved tensile properties to 1200 K and improved stress-rupture properties to 1100 K. For example, at 1000 K the zirconium-modified alloy exhibited a 40 percent higher yield strength and a four times greater stress-rupture life than the Ni-30Cu alloy.

In addition to an improved overall strength, it appeared that the grain boundaries in the zirconium-modified alloy were considerably strengthened in comparison to those in Ni-30Cu as the onset of grain boundary cracking was delayed to higher test temperatures. The oxidation characteristics of the modified alloy appeared to be equivalent to those of the base Ni-30Cr alloy.

INTRODUCTION

The Monel alloy 400, nominally Ni-30Cu, is used in moderate temperature applications where good corrosion resistance is required. In general, Monel 400 is not used in high temperature environments because of its low mechanical strength and poor oxidation resistance at temperatures above about 800 K. However, recent work (refs. 1 to 3) has identified Monel 400 as a suitable catalyst for the reduction of nitrogen oxide (NO_x) from internal combustion engines. In this application Monel operates at temperatures from nominally 975 to 1200 K, and it plays an active role in the NO_x reduction process as the

Monel is continuously subjected to oxidation and reduction reactions. One major problem associated with the use of Monel 400 in catalytic reactors has been the lack of long-term durability. Data from reference 2 indicate that the durability is related to grain boundary degradation and subsequent loss in strength. Thus, a higher strength Monel-type alloy with improved grain boundary strength and stability is desirable for this application.

As the oxidation behavior of the Monel alloy is important in catalytic applications, any attempt to strengthen the base alloy must not affect the overall surface oxidation characteristics. This requirement probably prohibits using the high strength aluminum-modified Monel alloy K-500 (nominally Ni-30Cu-3Al) as a continuous, unreduceable alumina scale could be formed. One possible method of strengthening Monel without affecting the oxidation behavior would be to introduce a low volume fraction of inert particles or precipitates into the alloy matrix. To this end, an attempt was made to strengthen a nominally 70Ni-30Cu alloy by slight alloying additions of zirconium followed by a hydrogen anneal. This procedure was designed to introduce a dispersion of ZrH_2 particles in the matrix in the same manner that ZrH_2 precipitates are formed in Mg-0.5Zr alloys (refs. 4 and 5). While precipitates were formed in the zirconium-modified Monel-type alloy, the precipitates were not ZrH_2 , as intended, but rather an intermetallic compound. However, the precipitates did increase the high temperature strength without affecting the oxidation characteristics of the Monel-base alloy.

This report describes the results of an exploratory study where a thermomechanical processing schedule is developed to precipitation harden a zirconium-modified Monel alloy. Also, the mechanical properties and oxidation characteristics of this alloy (Ni-30Cu-Zr) are compared to those of a base Monel alloy (Ni-30Cu).

The problems associated with using Monel alloys for catalytic reactors were brought to the author's attention by Dr. M. A. Dayananda of Purdue University.

EXPERIMENTAL

Alloy Preparation

Two Monel-type alloys of nominal composition, Ni-30Cu and Ni-30Cu-0.2Zr, were vacuum melted in alumina crucibles and cast into nominal 8 by 8 by 1.5 centimeter sheet-bar molds. The sheet-bar ingots were hot rolled in air in one direction at 1450 K from 1.5 to 0.4 centimeter and warm rolled in air in the same direction at 920 K from 0.4 to nominally 0.15 centimeter. Both the hot rolling and warm rolling schedules incorporated 10 percent reductions per pass followed by a 0.17-hour anneal at the rolling temperature. Chemical analyses of the as-processed alloys are given in table I.

Processing Studies

After rolling to gage both alloys were given a 0.5-hour anneal at 1365 K in hydrogen to promote recrystallization and grain growth to a reasonably large grain size (about 100 μm). After this anneal the alloys were subjected to several thermomechanical processing (TMP) treatments to determine a suitable processing schedule. In general, the TMP involved heat treatments at 1225, 1125, or 1025 K in hydrogen of as-annealed specimens and annealed plus 10 percent cold-worked (rolling at ambient temperature) specimens. The results of TMP as determined by hardness testing are given in table II. These data suggest that for each TMP schedule the Ni-30Cu-Zr alloy is stronger than the Ni-30Cu alloy and that the 10 percent cold work plus 1125 K heat treatment would yield the best strength improvement. Metallography of the TMP alloys revealed that the alloys had not recrystallized and that only the Ni-30Cu-Zr alloy specimens contained precipitates. A typical example of the precipitates found in the Ni-30Cu-Zr alloys is shown in figure 1. The grain size was determined by standard line intercept techniques, and the data are reported in table III. As the grain size appeared to be independent of the final heat treatment temperature, the reported grain size data are an average for the three final heat treatment temperatures.

On the basis of the metallography and hardness tests, the following TMP schedule was selected for further study: (1) 0.5-hour anneal at 1365 K in hydrogen, (2) approximately 10 percent cold work by ambient temperature rolling, and (3) final heat treatment at 1125 K in hydrogen. Metallography of the alloys subjected to this TMP schedule revealed some areas of small recrystallized grains in both alloys. In general, these areas were confined to the central region of the sheet thickness. The grain size, exclusive of the recrystallized areas, was determined to be 160 micrometers for the Ni-30Cu-Zr alloy and 115 micrometers for Ni-30Cu. These values are in agreement with those reported in table III. On the basis of the stress-rupture life and creep data for Monel reported in references 6 and 7, the small difference in grain size between these Ni-30Cu and Ni-30Cu-Zr alloys should have little effect on the elevated temperature, long-term mechanical properties.

Alloy Evaluation

Both Monel-type alloys were subjected to the previous TMP schedule with a 1.5-hour final anneal at 1125 K to ensure complete precipitation of the second phase in the Ni-30Cu-Zr alloy. Tensile-type specimens with a 2.54 by 0.63 centimeter gage section were blanked from the thermomechanically processed alloy sheet. In all cases, the gage length was parallel to the sheet rolling direction. Hardness tests conducted on the

blanking scrap revealed that the hardnesses (Rockwell F scale) of the Ni-30Cu and Ni-30Cu-Zr alloys were 85 and 95, respectively. The reason for the difference in hardness between the initial studies and scale-up could be due in part to possible overaging of the precipitates and regions of recrystallization.

Both Monel-type alloys were subjected to tensile testing in air at ambient temperature, 800, 1000, 1200, and 1400 K and to constant-load stress-rupture testing in air at 800, 1000, 1100, and 1200 K. In addition, several stress-rupture tests were interrupted prior to failure, and these specimens were tensile tested at ambient temperature to obtain a measure of creep damage. All mechanical property testing was conducted in accordance with ASTM standards. An additional characterization of the Monel-type alloys was the identification of the precipitates in the Ni-30Cu-Zr alloy and the oxides formed during elevated temperature testing.

RESULTS AND DISCUSSION

Identification of Precipitates

As the intention of this work was to strengthen the Monel-type alloy matrix with ZrH_2 particles, samples of both alloys after TMP were submitted for hydrogen analysis. The results of these analyses revealed that both the zirconium-modified alloy and the base Ni-30Cu alloy contained about 5 ppm hydrogen. Therefore, it was concluded that the precipitates in the zirconium-modified alloy were not ZrH_2 . In a further effort to identify the composition of precipitates and to study the distribution of precipitates, samples of both alloys were examined by electron microscopy techniques. Precipitates were observed only in the Ni-30Cu-Zr alloys; typical electron replica photomicrographs of the Ni-30Cu-Zr alloys are shown in figure 2. Platelet and needle type precipitates were observed within the grains and grain boundaries for both TMP schedules. The TMP schedule involving cold work resulted in finer precipitates and a corresponding better distribution than the TMP schedule without cold work.

Because of the larger precipitates, the unworked zirconium-modified alloy was used for particle identification. Both X-ray diffraction following a long chemical extraction (0.17 hr in 85 percent H_2O - 5 percent acetic acid - 10 percent HNO_3 solution) and electron diffraction following a short electrolytic extraction (0.02 hr in 90 percent H_2O - 10 percent H_3PO_4 solution at 15 V) on a carbon film identified the precipitates as a Ni_5Zr compound. Further examination of extracted precipitates with EDS analyzer on a SEM indicated that the precipitates contained only nickel and zirconium. Therefore, the strengthening agent in the zirconium-modified Monel-type alloy appears to be Ni_5Zr . This is somewhat surprising as Elliott (ref. 8) reports the solubility of zirconium in nickel to be about 0.9 percent between 1125 and 925 K. Apparently the presence of 30 per-

cent copper in the nickel solid solution severely reduces the solubility of zirconium.

Tensile Properties

The results of the room temperature and elevated temperature tensile tests are given in table IV, and the average strength properties are plotted in figure 3. These data indicate that the precipitates in the zirconium-modified alloy have improved the tensile properties, particularly between 800 and 1200 K where both strength and ductility improvements are apparent. For example, at 1000 K the 0.2 yield strength of the zirconium-modified alloy exceeds the ultimate tensile strength of the base Ni-30Cu alloy.

Examination of the microstructure of the tensile tested alloys indicates that grain boundary cracking occurred in all elevated temperature tests ($T \geq 800$ K) of the Ni-30Cu alloy while grain boundary cracking was only seen in the 1200 and 1400 K tensile tests of the zirconium-modified alloy. Typical photomicrographs of the fracture areas of the 1000 K tensile test specimens are shown in figure 4. From this figure it can be seen that the Ni-30Cu-Zr alloy failed in a ductile manner while the Ni-30Cu failed by grain boundary cracking. Thus, it appears that the precipitates in the zirconium-modified alloy have strengthened the grain boundaries at elevated temperatures.

Metallography also revealed that the room temperature tensile failure of both alloys occurred by ductile mechanisms, and failure at 1400 K for both alloys appeared to be the result of massive oxidation of alloy and grain boundary cracking. Oxidation of the base metal did not appear to affect the results of the tensile tests conducted at or below 1200 K as only thin oxide coatings were observed.

Stress-Rupture Properties

Stress-rupture tests of the Monel-type alloys were conducted in air at stress levels nominally designed to produce failure of the Ni-30Cu alloy in 100 hours. In general, testing was interrupted if the time under stress/temperature conditions exceeded 500 hours or if data from other tests indicated that the life expectancy would greatly exceed 500 hours. Specimen from the interrupted tests were then tensile tested at room temperature to obtain a measure of the amount of creep damage.

The results of the stress-rupture testing are given in table V. For the various stress/temperature conditions between 800 and 1100 K, the zirconium-modified alloy exhibited better properties than the Ni-30Cu alloy. In this temperature regime, the life of the Ni-30Cu-Zr alloy exceeded the life of the Ni-30Cu by at least a factor of four. Metallography of the ruptured specimens revealed that failure of the Ni-30Cu alloy was

probably due to grain boundary cracking at 800 K and a combination of grain boundary cracking and oxidation of the crack surfaces at 1000 and 1100 K. Examples of the latter type of failure can be seen in figure 5. Grain boundary cracks were also seen in the Ni-30Cu-Zr specimen which failed at 800 K. Failure of Ni-30Cu-Zr specimens at 1100 K appeared to be the result of grain boundary cracks and oxidation at the cracks; however, as can be seen in figure 5, the overall damage to the microstructure after testing at 1100 K does not appear to be as severe in the Ni-30Cu-Zr alloy as in the Ni-30Cu alloy.

Testing either Monel-type alloy at 10 meganewtons per square meter (MN/m^2) and 1200 K resulted in completely oxidized cross sections. Thus, the strength improvement of the zirconium-modified alloy at 1200 K, as indicated by tensile testing, cannot be realized in a highly oxidizing atmosphere.

Residual Tensile Properties

The residual room temperature tensile properties of interrupted stress-rupture tested specimens are given in table VI. The zirconium-modified alloy exhibited superior residual tensile properties when compared to the Ni-30Cr alloy. For example, at similar exposure conditions of $21 MN/m^2$, 1000 K, and 362 hours the Ni-30Cu alloy exhibited a severe reduction in ultimate tensile strength and tensile elongation while the Ni-30Cu-Zr alloy retained tensile properties which are essentially equivalent to those of the unexposed Ni-30Cu alloy.

The data in table VI for the exposed and unexposed Ni-30Cu specimens indicate that a long-time exposure at 1000 K under a low stress ($15 MN/m^2$) has only a moderate effect on the residual room temperature tensile strength properties of this alloy. On the other hand, exposure to a slightly higher stress ($21 MN/m^2$) at 1000 K tends to drastically reduce the residual ultimate tensile strength and tensile elongation (although the yield strength was not as greatly affected). The microstructure of the tensile-tested specimens revealed that the specimen exposed for 361 hours at $15 MN/m^2$ and 1000 K had only a few grain boundary cracks and failed in a ductile manner; the specimens exposed to $21 MN/m^2$ and 1000 K for 171 and 362 hours had many grain boundary cracks which severely reduced the effective load-bearing area. Apparently tensile fracture in the latter specimens occurred by ductile failure of the matrix alloy between adjacent grain boundary cracks. While ductile fracture may indeed take place in localized regions of the specimens previously exposed to 1000 K and $21 MN/m^2$ conditions, the overall tensile failure would be considered brittle because of the low ductility.

The residual property data in table VI for the Ni-30Cu-Zr alloy indicate that prior exposure can also affect the tensile properties of this alloy. Comparison of the properties of the unexposed alloy to the properties of Ni-30Cu-Zr subjected to $131 MN/m^2$ and 800 K for 505 hours reveals that this exposure decreased the ductility somewhat and

possibly increased the strength properties. After exposure at 1000 K, either at 21 MN/m² for 362 hours or 35 MN/m² for 501 hours, the tensile ductility of the exposed alloys is nearly equivalent to that of the unexposed material; however, exposure at 1000 K did reduce the strength properties. After 362 hours at 21 MN/m² and 1000 K, the 0.2 yield strength was about 17 percent less and the ultimate tensile strength was about 10 percent lower than the unexposed alloy values. On the other hand, the residual strength properties of Ni-30Cu-Zr exposed at 21 MN/m² and 1000 K for 362 hours are nearly comparable to the unexposed Ni-30Cu alloy; Ni-30Cu subjected to a similar exposure exhibited severely reduced residual properties (a 20 percent reduction in yield strength and a 50 percent reduction in ultimate tensile strength). The Ni-30Cu-Zr specimens exposed to 35 MN/m² and 1000 K for 501 hours suffered about a 30 percent reduction in 0.2 yield strength and about a 20 percent reduction in ultimate tensile strength when compared to the unexposed alloy. In addition, the residual strength properties of the Ni-30Cu-Zr alloy exposed for 501 hours at 35 MN/m² and 1000 K are about 40 MN/m² lower than those of unexposed Ni-30Cu. However, the Ni-30Cu specimens failed in a relatively short time (~100 hr) when tested under these conditions. Thus, in effect, the greater initial strength of the zirconium-modified alloy prolongs the usable load-carrying life of the Ni-30Cu alloy under high-temperature oxidizing conditions.

Metallographic examination of the Ni-30Cu-Zr residual property specimens revealed similar microstructures for all exposure conditions. Internal grain boundary cracks were not observed; however, grain boundary cracks emanating from the sheet surfaces were seen. In addition, all tensile fractures appeared to be ductile, as illustrated by the microstructure in figure 6.

Overall, the precipitate-strengthened Monel-type alloy has much better stress-rupture properties and resistance to creep damage than the Ni-30Cu alloy. Apparently the precipitates in Ni-30Cu-Zr strengthen the grain boundaries which leads to the improved elevated temperature characteristics.

Composition of Oxide Scale

An X-ray analysis of the surface oxides formed during stress-rupture testing of Ni-30Cu and Ni-30Cu-Zr alloys at 800 and 1100 K indicated the presence of both CuO and NiO. The presence or absence of Cu₂O could not be confirmed as Cu₂O X-ray diffraction lines overlap those of NiO. Zirconium oxides were not detected in the oxide scale formed on the Ni-30Cu-Zr specimens. In general, the oxide scales formed on Ni-30Cu and Ni-30Cu-Zr were identical.

CONCLUSIONS

A precipitation-strengthened Monel-type alloy has been developed by adding minor amounts of zirconium to a base Ni-30Cu composition. The strengthening agent in the modified alloy has been tentatively identified as a Ni₅Zr compound. Precipitation strengthening resulted in improved tensile properties to 1200 K and stress-rupture properties to 1100 K. For example, at 1000 K the zirconium-modified alloy exhibited a 40 percent higher yield strength and a four times greater stress-rupture life than a Ni-30Cu alloy. In addition to an improved overall strength, it appeared that the grain boundaries in the zirconium-modified alloy were considerably strengthened in comparison to those in Ni-30Cu as the onset of grain boundary cracking was delayed to higher test temperatures. The improved mechanical properties coupled with the observation that identical oxide scales were formed on the zirconium-modified alloy and the base Ni-30Cu alloy seem to indicate that the zirconium-modified Monel-type alloy might have potential for use as a catalyst for the reduction of nitrogen oxides from internal combustion engines.

Lewis Research Center ,

National Aeronautics and Space Administration,

Cleveland, Ohio, March 22, 1974,

501-21.

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TABLE I. - CHEMICAL ANALYSIS

MONEL-TYPE ALLOYS

Alloy	Composition, wt %		
	Cu	Zr	Ni
Ni-30Cu	29.6	---	Balance ^a
Ni-30Cu-Zr	29.3	0.2	Balance ^a

^aSpectrographic analysis revealed faint traces of Al, Co, Cr, Fe, Mg, Mn, and Ti.

TABLE II. - HARDNESS OF THERMOMECHANICAL PROCESSED

MONEL-TYPE ALLOYS

[Rockwell F scale; 0.16-cm ball; 60-kg load; starting condition for both alloys, 1/2-hr anneal at 1365 K.]

Alloy	Temperature of final heat treatment, K	0 Percent cold work prior to final heat treatment		10 Percent cold work (rolling) prior to final heat treatment	
		Time of final heat treatment, hr	Rockwell F hardness	Time of final heat treatment, hr	Rockwell F hardness
Ni-30Cu	None	--	75	---	^a 101
Ni-30Cu-Zr	None	--	77	---	^a 102
Ni-30Cu	1225	1	75	1/2	76
Ni-30Cu-Zr	1225	1	84	1/2	93
Ni-30Cu	1125	2	77	1/2	73
Ni-30Cu-Zr	1125	2	89	1/2	103
Ni-30Cu	1025	3	77	1/2	96
Ni-30Cu-Zr	1025	3	88	1/2	101

^aExtrapolated from Rockwell B scale readings.

TABLE IV. - TENSILE PROPERTIES OF MONEL-TYPE ALLOYS

[Room temperature tests: cross head speed, 0.0127 cm/min until ~0.6 percent strain and then 1.27 cm/min until fracture. Elevated temperature tests: cross head speed, 0.127 cm/min.]

Composition	Temperature, K	0.2 Percent yield stress, MN/m ²	Ultimate tensile strength, MN/m ²	Elongation, percent
Ni-30Cu	Room	200	405	45
		215	427	43
		186	411	45
		202	416	45
Ni-30Cu-Zr		199	415	47
		254	482	35
		234	455	36
		242	471	37
Ni-30Cu	800	164	261	16
		170	260	24
Ni-30Cu-Zr		216	390	28
		202	374	32
Ni-30Cu	1000	108	138	7
		93	136	18
Ni-30Cu-Zr		146	178	24
		143	182	24
Ni-30Cu	1200	63	69	20
		63	68	23
Ni-30Cu-Zr		77	83	28
		78	87	30
Ni-30Cu	1400	22	22	40
Ni-30Cu-Zr	1400	22	22	--

TABLE III. - AVERAGE GRAIN DIAMETER FOR
THERMOMECHANICAL PROCESSED
MONEL-TYPE ALLOYS

Alloy	Grain diameter, μ m	
	No cold work prior to final heat treatment	10-Percent cold work prior to final heat treatment
Ni-30Cu	115	95
Ni-30Cu-Zr	185	160

TABLE V. - STRESS-RUPTURE PROPERTIES OF
MONEL-TYPE ALLOYS

Composition	Test condition		Life, hr	Elongation percent
	Temperature, K	Stress, MN/m ²		
Ni-30Cu	800	138	74.9	10
			114.5	7
Ni-30Cu-Zr			^a 504.5	~1
		159	^a 504.5	~1
Ni-30Cu	1000	15	^a 361.4	2
		21	^a 170.5	2
Ni-30Cu-Zr		21	^a 362.2	4
		35	56.7	7
Ni-30Cu	1100	35	153.0	17
		21	^a 362.2	~1
Ni-30Cu-Zr		35	^a 500.9	~1
		35	^a 500.9	~1
Ni-30Cu		21	28.1	7
			71.8	18
Ni-30Cu-Zr			238.2	9
		173.8		6
Ni-30Cu	1200	10	95.5	Almost completely oxidized test sections
			108.5	
Ni-30Cu-Zr			107.3	
		108.7		

^aSpecimen removed prior to failure.

TABLE VI. - RESIDUAL ROOM TEMPERATURE TENSILE PROPERTIES OF
EXPOSED MONEL-TYPE ALLOYS

Alloy	Prior exposure			Offset 0.2 yield stress, MN/m ²	Tensile properties ^a	
	Stress, MN/m ²	Temperature, K	Life, hr		Ultimate tensile strength, MN/m ²	Elongation, percent
Ni-30Cu	None	None	---	^b 200	^b 414	^b 44
	15	1000	361	145	360	40
	21	1000	171	162	300	8
	21	1000	362	163	202	5
Ni-30Cu-Zr	None	None	None	^b 243	^b 469	^b 36
	138	800	505	278	503	30
	138	800	505	265	480	23
	21	1000	362	200	426	33
	35	1000	501	164	365	35
	35	1000	501	168	379	36

^aStrength properties based on original cross-sectional area.

^bAverage properties.

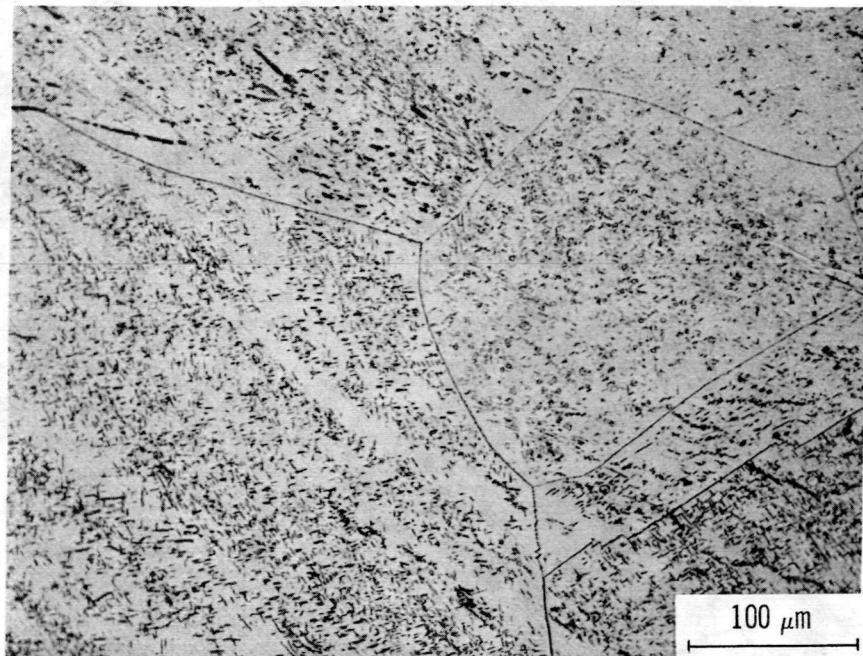
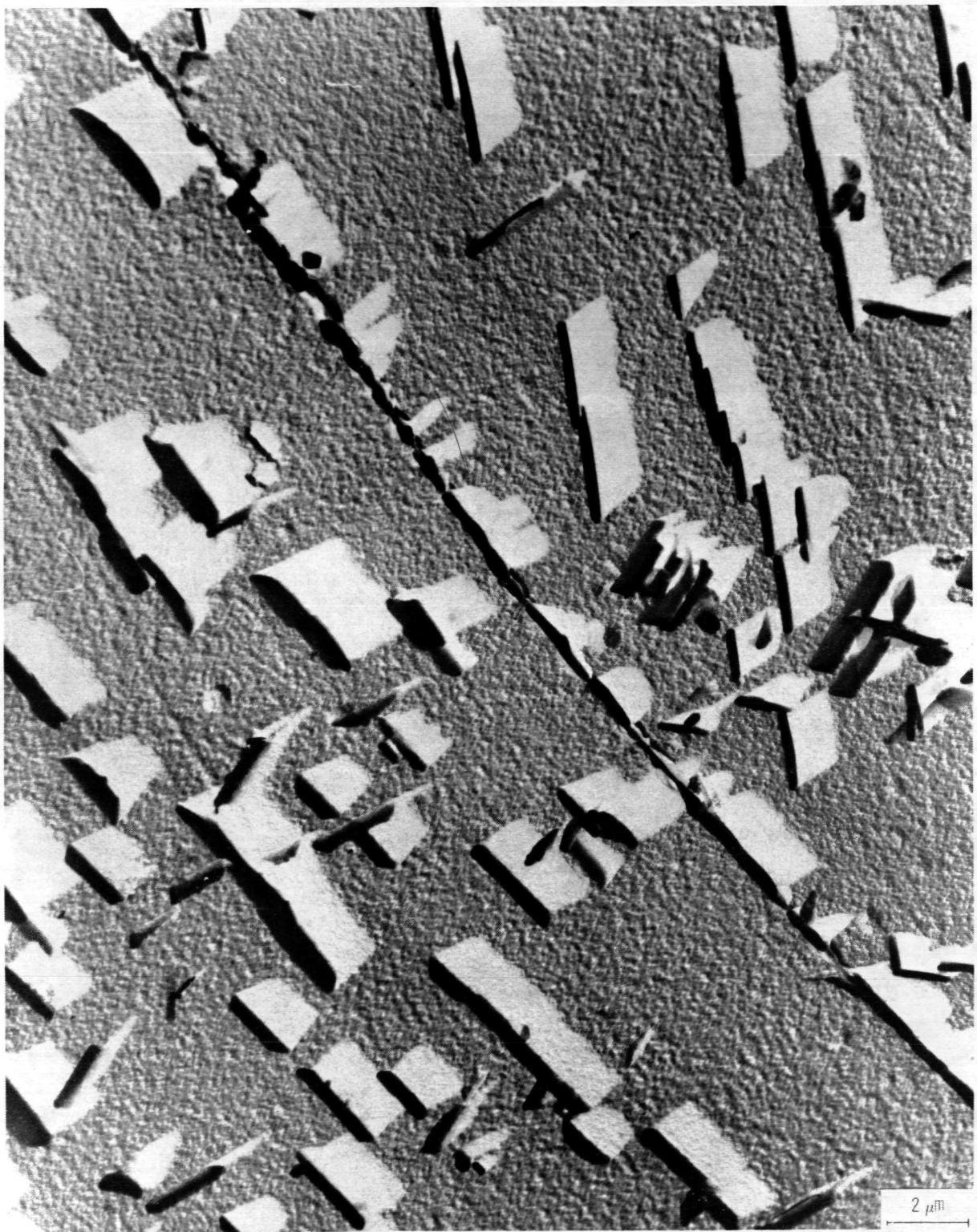
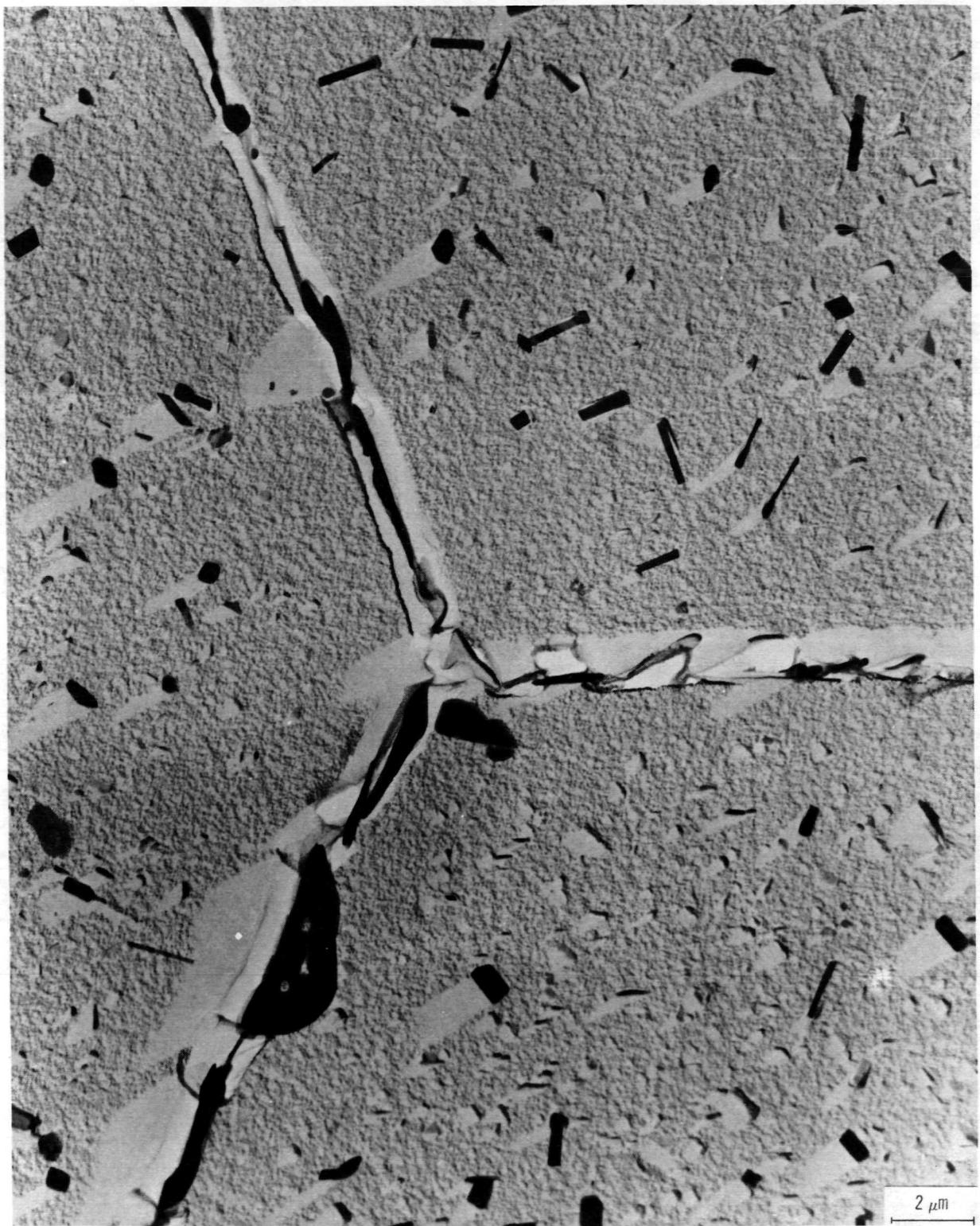


Figure 1. - Typical microstructure of Ni-30Cu-Zr alloy annealed 0.5 hour at 1365 K and then 2 hours at 1125 K in hydrogen. Electrolytically etched with 30HNO₃-30H₂O-30 glycerin.



(a) Annealed 0.5 hour at 1365 K and then 2 hours at 1125 K.

Figure 2. - Electron replica photomicrographs of Ni-30Cu-Zr.



(b) Annealed 0.5 hour at 1365 K, cold worked 10 percent, and then annealed 1.5 hours at 1125 K.

Figure 2. - Concluded.

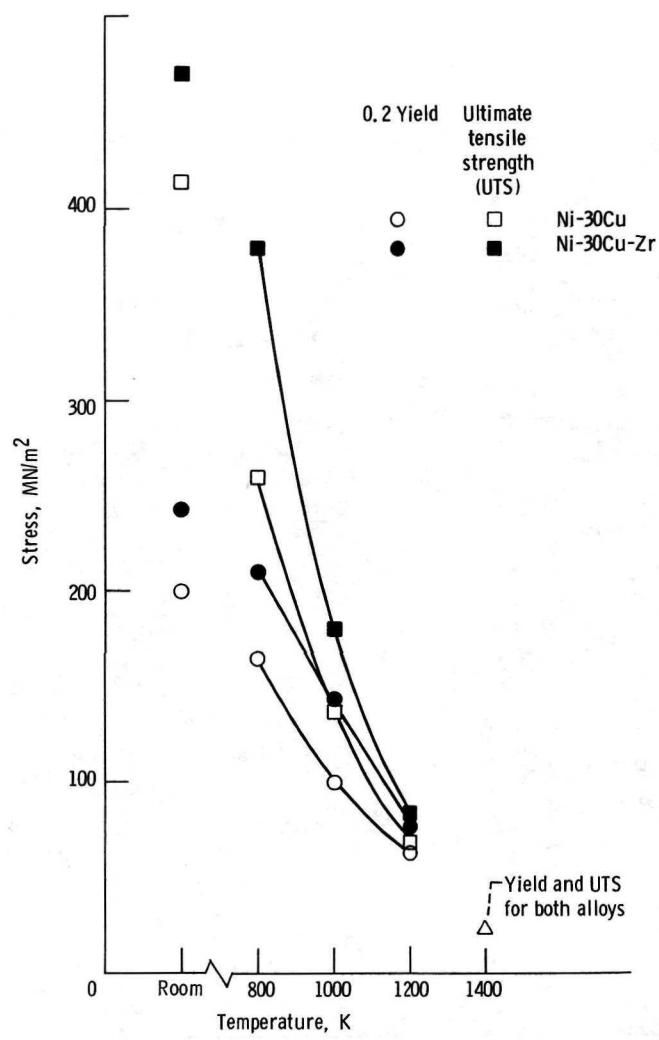
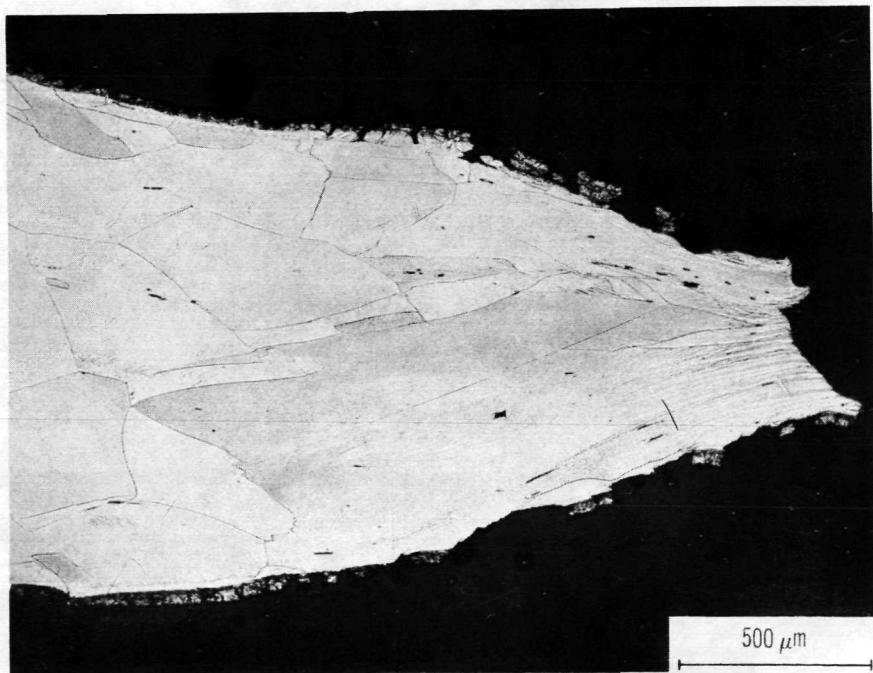


Figure 3. - Average tensile strength properties as function of temperature for Monel-type alloys.

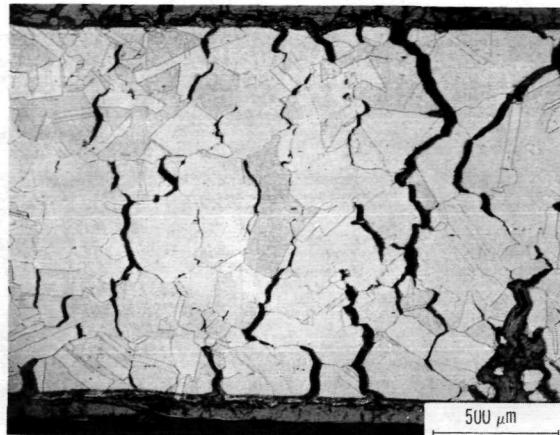


(a) Ni-30Cu, 7 percent elongation.

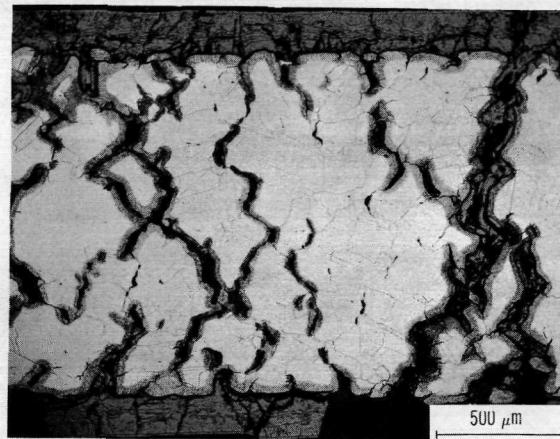


(b) Ni-30Cu-Zr, 24 percent elongation.

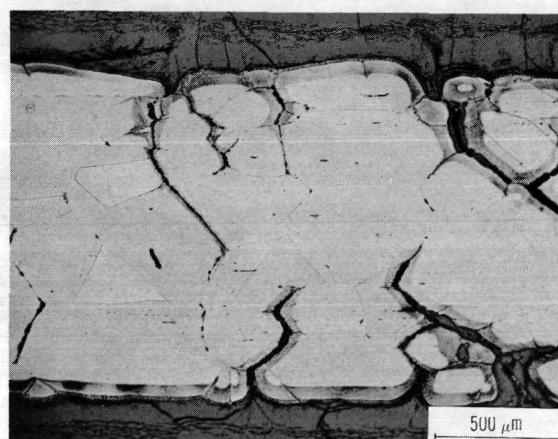
Figure 4. - Typical photomicrographs of fracture regions of 1000 K tensile test specimens of Monel-type alloys. Electrolytically etched with $30\text{HNO}_3\text{-}30\text{H}_2\text{O}\text{-}30\text{glycerin}$.



(a) Alloy, Ni-30Cu; temperature, 1000 K; stress, 35 MN/m²; life, 153 hours; elongation, 17 percent.



(b) Alloy, Ni-30Cu; temperature, 1100 K; stress, 21 MN/m²; life, 71.8 hours; elongation, 18 percent.



(c) Alloy, Ni-30Cu-Zr; temperature, 1100 K; stress, 21 MN/m²; life, 238.2 hours; elongation, 9 percent.

Figure 5. - Typical microstructures of stress-rupture tested Monel-type alloys. Electrolytically etched with 3UHNO₃-30H₂O-30 glycerin.

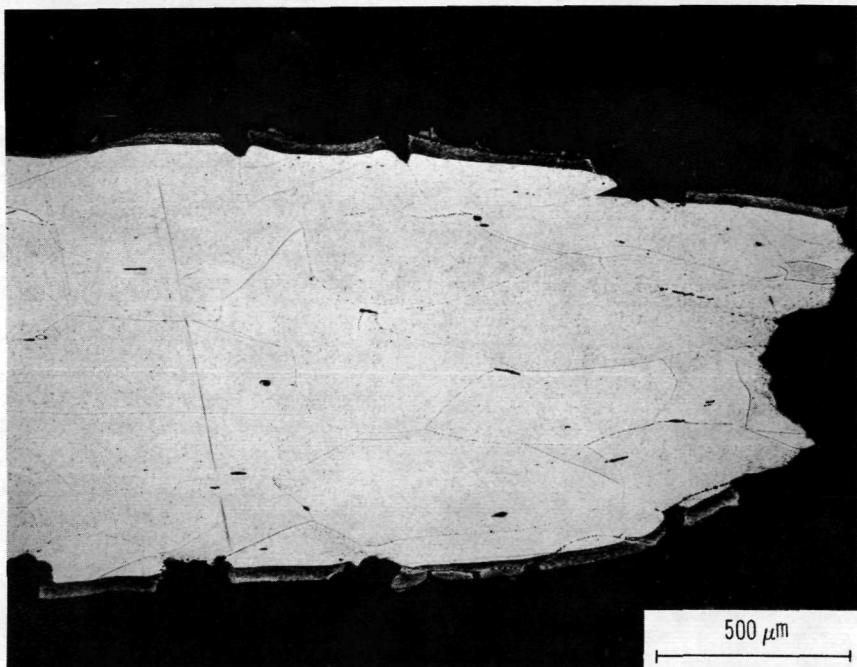


Figure 6. - Tensile fracture region of Ni-30Cu-Zr specimen after 501-hour exposure to 1000 K at 35 MN/m². Electrolytically etched with 30HNO₃-30H₂O-30 glycerin.

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